

FIELD ION MICROSCOPY AND ATOM PROBE TOMOGRAPHY OF METAMORPHIC MAGNETITE CRYSTALS

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Atom Probe Tomography (APT) is a highly attractive technique for the nanoanalysis of geological materials despite the difficulties inherent in analyzing semi-conducting and insulating materials. A schematic diagram of the physics of the three-dimensional atom probe (3DAP) is shown in Figure 1 [1].

Field ion specimens have been successfully fabricated from samples of metamorphic magnetite crystals (Fe_3O_4) extracted from a polymetamorphosed, granulite-facies marble with the use of a focused ion beam (FIB) [2]. This particular magnetite was chosen for several reasons. First, magnetite is a common mineral on Earth, the Moon and Mars. Second, magnetite is one of the more conductive of the minerals, having a resistivity of 52×10^{-4} ohm-cm [3]. Finally, this particular magnetite contains disk-shaped exsolutions approximately 40 nm in diameter, 1-3 nm thick and about 10^4 platelets/ μm^3 which have been shown by qualitative energy-dispersive X-ray spectroscopy (EDX) to contain elevated concentrations of manganese and aluminum [3,4]. These lamellae make this magnetite particularly attractive for investigating the capabilities of 3DAP for the nanoanalysis of geological materials.

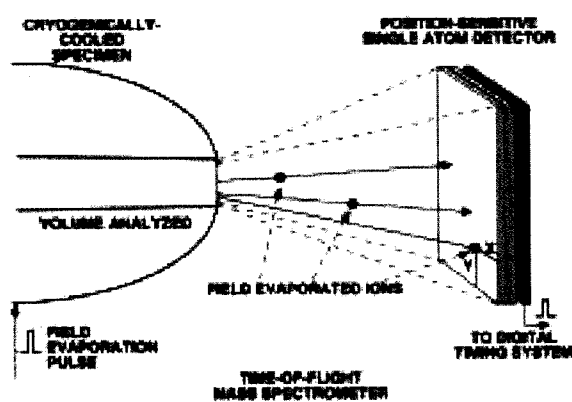


Figure 1: Schematic diagram of APT nanoanalysis [1].

Field ion microscope (FIM) images of these magnetite crystals were obtained in which the observed size and morphology of the precipitates agree with previous results (Figure 3). This field ion micrograph reveals precipitates as thin dark bands. Two distinct {111} crystallographic poles are clearly seen. Two of the precipitates seen in the FIM image, Figure 3, were

also visible in the TEM dark field image, Figure 4, taken after field ion evaporation.



Figure 2: Focused ion beam induced, secondary electron images of magnetite sample 031300E. a) Sample as mounted with Pt deposition at the base to improve electrical connection. b) Sample after extensive ion milling using various beam currents.

A limited number (<3000) of ions was collected in the energy compensated optical position-sensitive atom probe (ECOPoSAP), peaks for singly ionized ^{16}O , ^{56}Fe ,

and $^{56}\text{Fe}^{16}\text{O}$ and doubly ionized $^{16}\text{O}_2^{2+}$, $^{54}\text{Fe}^{2+}$, $^{56}\text{Fe}^{2+}$ and $^{57}\text{Fe}^{2+}$ peaks were fully resolved. Occasionally two or more atoms field evaporate together as a (molecular) ion such as FeO or O_2 . A depletion in the amount of total oxygen measured versus the stoichiometric amount expected was observed (Figure 4). Manganese and aluminum were also observed in a limited analysis of a single lamella in the ECOPoSAP.

References: [1] Miller, M. K. (2000) *Atom Probe Tomography: Analysis at the Atomic Level* (Kluwer Academic/Plenum Publishers, New York), [2] Kuhlman, K. R. *et al.* (2001) accepted by *Ultramicroscopy*, [3] Sitzman, S. D. *et al.* (2000), *American Mineralogist*, **85**(1) 14-21, [4] Valley, J. W. and C. M. Graham (1993), **259**, 1729-1733.

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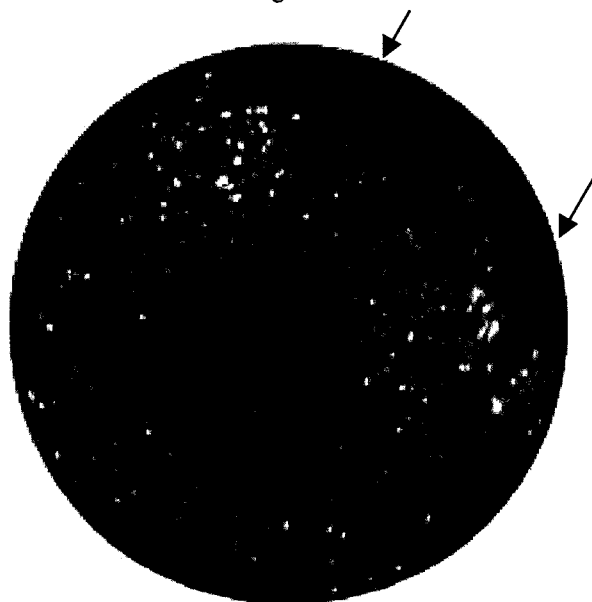


Figure 3: Field ion micrograph of magnetite LP204-1 and oriented precipitates (indicated by arrows) at approximately 16 kV. Note the clarity of the $\{111\}$ poles and the precipitate running directly through the pole at right of the image.

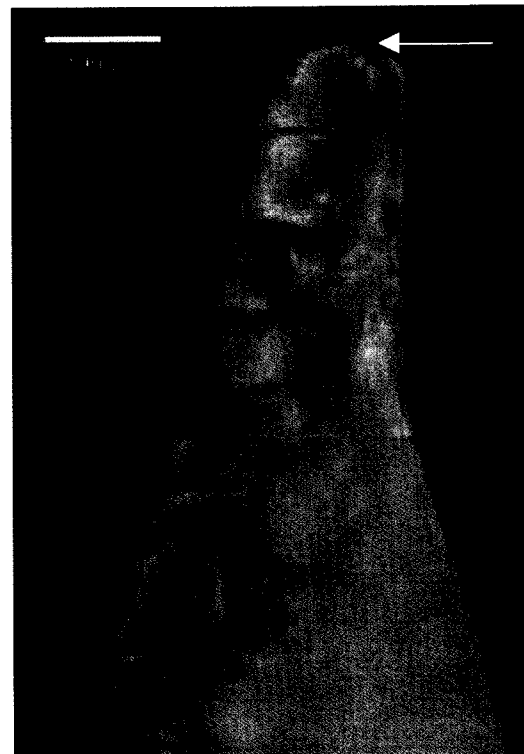


Figure 4: Transmission electron micrograph of magnetite sample after FIM analysis. The precipitates indicated by the arrow at the very tip are the same precipitates seen in Figure 3.

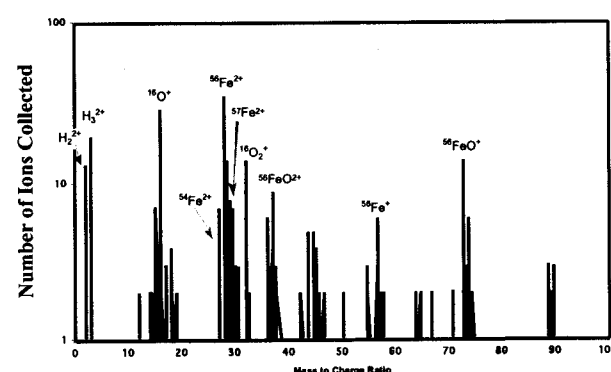


Figure 5: Preliminary mass spectrum of the matrix in magnetite, LP204-1. This spectrum contains 2267 atoms.